



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



Publication number: **0 657 104 A1**

12

## EUROPEAN PATENT APPLICATION

21 Application number: 93203463.0

51 Int. Cl.<sup>8</sup>: A23D 7/00

22 Date of filing: 09.12.93

43 Date of publication of application:  
14.06.95 Bulletin 95/24

54 Designated Contracting States:  
AT BE CH DE DK ES FR GB GR IE IT LI LU MC  
NL PT SE

71 Applicant: UNILEVER N.V.  
P.O. Box 760  
NL-3000 DK Rotterdam (NL)

72 Inventor: Leser, Martin E. Unilever Res.  
Vlaardingen Lab.  
O.van Noortlaan 120  
NL-3133 AT Vlaardingen (NL)

74 Representative: Keppels, Willem Richard  
Engelbertus Gerardus, Drs. et al  
Unilever N.V.  
Patent Division  
P.O.Box 137  
NL-3130 AC Vlaardingen (NL)

54 Edible Water-In-Oil Microemulsions.

57 Thermodynamically stable transparent edible water-in-oil microemulsion comprising up to 33% of water, 0.1-30% of a diacylglyceride having one strong polar hydrophilic group, 0.1-30% of a monoacylglyceride and oil.

EP 0 657 104 A1

This invention relates to an edible water-in-oil microemulsion.

For incorporating water and polar ingredients into edible oils emulsions can be used, usually resulting in cloudy or opaque products, although stable, clear liquids can be obtained by making microemulsions using a well selected combination of oils, polar liquids and surfactants. US-A-5,045,337 describes edible W/O microemulsions using polyglycerolesters as surfactants, whereby up to 5% water can be incorporated into the microemulsion, although in the examples only 1% water is used. Attempts to incorporate more water resulted in not completely stable microemulsions. It would be desirable to make more stable microemulsions, having the possibility of incorporating more water and/or other polar ingredients and to use other surfactants being generally acceptable in the food industry.

The invention provides thereto a thermodynamically stable transparent edible water-in-oil microemulsion comprising one or more oils, oil-soluble surfactants and polar ingredients characterized by up to 33% water and a surfactant comprising 0.1-30% of a diacylglyceride selected from the group of phospholipids and diacylglycerideesters selected from the group of citrates, tartrates and lactilates and 0.1-30% of a monoacylglyceride. In general these diacylglycerides have a strong polar hydrophilic group.

Preferably the acylgroups in the surfactants are roughly of the same character as the bulk of the acyl groups present in the triacylglycerides forming the oil of the microemulsion.

Particularly preferred diacylglyceride surfactants being phospholipids and preferred monoacylglycerides being those having acylgroups with 8-18 carbon atoms. Monoacylglycerides having unsaturated acylchains are in particular preferred.

It does not need explanation that all ingredients of the emulsions should be edible. Similarly the oil or oil mixture should preferably be refined.

As stated before the microemulsions can in practice contain up to about 33% of water. When trying to increase this amount the emulsion turns turbid. The lower level of water cannot practically be defined: any amount of water over the amount which is soluble in the oil-surfactant mixture will result in a microemulsion on mixing.

Dependent on the amount of aqueous phase the amount of oil phase can vary from 20 to 99.8%.

In this specification and claims all percentages refer to weights unless otherwise indicated.

Both pure and naturally occurring mixtures of lecithins and modified lecithins can be used as phospholipids in the microemulsions of this invention.

The term "oil" in this specification and claims includes triacylglycerides (predominantly) and diacylglycerides of application. Consequently some solid fats may be dissolved therein. The origin may be both natural and synthetic. Natural oils and fats may be vegetable, animal or marine origin, although the first source is preferred.

Microemulsions can be favourably used for incorporating oil-insoluble but water soluble ingredients into oils without changing the optical character of said oils. Examples of such ingredients being salts, sugar, aminoacids, vitamin B, C and K, flavours, flavour precursors, proteins, flavour boosters (eg. 5'-nucleotides) trace minerals, organic acids, peptides, and water soluble anti-oxidants and usually water contents up to 5% of the emulsion will be sufficient for this purpose.

The following examples are merely illustrative of the present invention.

The general procedure was as follows: the water soluble ingredients were dissolved in the water for the microemulsion and the surfactants in the oil. Any undissolved material was separated by centrifuging or filtration. Thereafter the clear liquids are combined and thoroughly mixed using a mixer.

#### Example 1.

Refined sunflower oil (88,49) was mixed with 10 g of a surfactant consisting of 1 g glycerophosphatidylcholine (PC) and 9 g glyceromono-oleate (GMO). After obtaining a clear liquid 1.6 g water is added and mixing was continued until again a clear liquid was obtained. Instead of water an 1:1 aqueous solution of 1 M glycine and fructose could be used.

#### Example 2.

88 g refined corn oil was mixed with 1 g glycerol phosphatidylethanolamine and 9 g GMO. 2 g water could be incorporated in this mixture. The water could contain about 30 % NaCl.

#### Example 3.

The process of example 1 was repeated using 32,5 g medium chain triglyceride oil (chain lengths 8-10 carbon atoms), 15 g PC, 20 g glyceromonocaprylate and 32,5 g water.

#### Example 4.

The process of example 1 was repeated using 20 g groundnut oil, 20 g PC, 40 g GMO and 20 g water.

**Claims**

1. Thermodynamically stable transparent edible water-in-oil microemulsion comprising one or more oils, oil-soluble surfactants and polar ingredients characterized by up to 33% water and a surfactant comprising 0.1-30% of a diacylglyceride selected from the group of phospholipids and diacylglycerideesters selected from the group of citrates, tartrates, lactilates and 0.1-30% of a monoacylglyceride. 5 10
2. Microemulsion according to claim 1 wherein the acylgroups in the surfactants are roughly of the same character as the bulk of the acyl groups present in the triacylglycerides forming the oil of the microemulsion. 15
3. Microemulsion according to claim 1 or 2 wherein the diacylglyceride surfactants are phospholipids. 20
4. Microemulsion according to claim 1, 2 or 3, wherein the monoacylglycerides have acylgroups with 8-18 carbon atoms. 25
5. Microemulsion in accordance with any of claims 1-4, wherein the monoacylglycerides have unsaturated acylchains. 30
6. Microemulsion in accordance with any of claims 1-5, wherein the oil or oils are refined. 35

40

45

50

55

60



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 93 20 3463

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	WO-A-93 12664 (KARLSHAMNS OILS & FATS AB) * page 3, line 27 - page 4, line 23 * * example 3 * * examples 1,3,5 * ---	1,3,6	A23D7/00
A	MAGDA EL-NOKALY AND DONALD CORNELL 'ACS SYMPOSIUM SERIES 448 : MICROEMULSIONS AND EMULSIONS IN FOODS' 1991, AMERICAN CHEMICAL SOCIETY, WASHINGTON, DC, US * page 26, abstract * * page 29; table I * * page 32, paragraph 5 * * page 35, paragraph 2 -paragraph 3 * * page 36; table II * * page 37; table III.A. * * page 38, paragraph 1 * * page 39; figures 6,7 * ---	1,4-6	
A	JOURNAL OF DISPERSION SCIENCE AND TECHNOLOGY vol. 11, no. 5, 1990 pages 479 - 489 L. ENGSTRÖM 'Aggregation and structural changes in the L2-phase in the system water/soybean oil/sunflower oil monoglycerides' * the whole document * ---	1,4,5	TECHNICAL FIELDS SEARCHED (Int.Cl.6) A23D
A	JOURNAL OF DISPERSION SCIENCE AND TECHNOLOGY vol. 8, no. 3, 1987 pages 207 - 215 L. MOBERGER ET AL. 'A study of fat oxidation in a microemulsion system' * the whole document * --- -/--	1,4,5	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 3 May 1994	Examiner Dekeirel, M
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons A : member of the same patent family, corresponding document			



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 93 20 3463

DOCUMENTS CONSIDERED TO BE RELEVANT															
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)												
A	JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY. vol. 62, no. 9, 1985, CHAMPAIGN US pages 1379 - 1385 AINO-MAIJA VESALA ET AL. 'Increasing the stability of vegetable oil solutions with the aid of monoglycerides and a cosurfactant' * the whole document * ---	1													
D,A	US-A-5 045 337 (MAGDA EL-NOKALY ET AL.) * the whole document * ---	1													
A	EP-A-0 378 893 (KAO CORPORATION) * claims 1-10 * -----	1													
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)												
The present search report has been drawn up for all claims															
Place of search THE HAGUE		Date of completion of the search 3 May 1994	Examiner Dekeirel, M												
<table border="0"><tr><td>CATEGORY OF CITED DOCUMENTS</td><td></td></tr><tr><td>X : particularly relevant if taken alone</td><td>T : theory or principle underlying the invention</td></tr><tr><td>Y : particularly relevant if combined with another document of the same category</td><td>E : earlier patent document, but published on, or after the filing date</td></tr><tr><td>A : technological background</td><td>D : document cited in the application</td></tr><tr><td>O : non-written disclosure</td><td>L : document cited for other reasons</td></tr><tr><td>P : intermediate document</td><td>A : member of the same patent family, corresponding document</td></tr></table>				CATEGORY OF CITED DOCUMENTS		X : particularly relevant if taken alone	T : theory or principle underlying the invention	Y : particularly relevant if combined with another document of the same category	E : earlier patent document, but published on, or after the filing date	A : technological background	D : document cited in the application	O : non-written disclosure	L : document cited for other reasons	P : intermediate document	A : member of the same patent family, corresponding document
CATEGORY OF CITED DOCUMENTS															
X : particularly relevant if taken alone	T : theory or principle underlying the invention														
Y : particularly relevant if combined with another document of the same category	E : earlier patent document, but published on, or after the filing date														
A : technological background	D : document cited in the application														
O : non-written disclosure	L : document cited for other reasons														
P : intermediate document	A : member of the same patent family, corresponding document														

EPO FORM 1503 (01.92) (P/0001)